

## PATENT COOPERATION TREATY

PCT

## NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents  
United States Patent and Trademark  
Office  
Box PCT  
Washington, D.C.20231  
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 04 September 2000 (04.09.00)	
International application No. PCT/GB99/04353	Applicant's or agent's file reference
International filing date (day/month/year) 22 December 1999 (22.12.99)	Priority date (day/month/year) 22 December 1998 (22.12.98)
Applicant WELTON, Nicholas, Jason et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:

20 July 2000 (20.07.00)

☐ in a notice effecting later election filed with the International Bureau on:
2. The election ☒ was
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland  Facsimile No.: (41-22) 740.14.35	Authorized officer  Olivia TEFY  Telephone No.: (41-22) 338.83.38
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## PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

REC'D 05 APR 2001

PCT

Applicant's or agent's file reference CTE/PL62259WO	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/GB99/04353	International filing date (day/month/year) 22/12/1999	Priority date (day/month/year) 22/12/1998
International Patent Classification (IPC) or national classification and IPC C08L33/06		
Applicant H.B. FULLER COATINGS LTD. et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 4 sheets, including this cover sheet.

☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 5 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand  20/07/2000	Date of completion of this report  02.04.2001
Name and mailing address of the international preliminary examining authority:   European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer  Krail, G  Telephone No. +49 89 2399 8302  

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/GB99/04353

**I. Basis of the report**

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17):*

**Description, pages:**

1-19 as originally filed

**Claims, No.:**

1-23 with telefax of 26/02/2001

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).  
☐ the language of publication of the international application (under Rule 48.3(b)).  
☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.  
☐ filed together with the international application in computer readable form.  
☐ furnished subsequently to this Authority in written form.  
☐ furnished subsequently to this Authority in computer readable form.  
☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.  
☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:  
☐ the claims, Nos.:  
☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/GB99/04353

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Yes:	Claims	1-23
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-23
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-23
	No:	Claims	

2. Citations and explanations  
**see separate sheet**

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/GB99/04353

**R item V**

Reference is made to the following document/s/:

- ✓ D1: DATABASE WPI Section Ch, Week 7917 Derwent Publications Ltd., London, GB; Class A14, AN 79-32158B XP002104164 & JP 54.034359 A (MITSUBISHI RAYON CO LTD) , 13 March 1979 (1979-03-13)
- ✓ D2: RAZINSKAYA ET AL: 'Study of the synergism of fireproofing agents' PLAST MASSY, no. 2, 1983, pages 49-50, XP002104162
- ✓ D3: ARAKAWA, ABE, IMAI: 'Effect of fire-retardant elements' KOBUNSHI RONBUNSHU (JAPANESE POLYMER SCIENCE AND TECHNOLOGY), vol. 31, no. 10, 1974, pages 643-7, XP002104163 TOKYO JP

D1 discloses a polymer composition comprising a poly methyl methacrylate, a halogen donor compound ,  $Sb_2O_3$  a "halogen volatilisation agent" and glass fibre and talc. The subject matter of claim 1 differs therefrom in that a (further specified) mixture of two poly acrylates is used and in that a char inducing component is present.

D1 refers to another technical field, namely to electrical materials.

D2 discloses such a blend of poly methyl methacrylate, PVC,  $Sb_2O_3$  and triethyl phosphate (cf. e.g. the first table);

D3 discloses blends of acrylic polymer, a "halogen volatilisation agent" (Geon, PVC),  $Sb_2O_3$  ("halogen volatilisation agent") and triethyl phosphate.

However, with both citations the composition does not include a char inducing additive, nor is there an indication that the polyacrylate is of such a nature that makes it suitable as a capstock polymer.

The citations do not refer to capstock polymers; even in combination (if the combination were justified) the do not give incentives to the combination of features in claim 1.

Thus, the composition of claim 1 and the laminate according to claim 10 are novel (Art. 33(2) PCT) and inventive (Art. 33(3) PCT) .

Industrial applicability (Art 33(4) PCT) is given.

CLAIMS:

1. A capstock polymer composition comprising a blend of:  
a melt extrudable acrylic polymer component;  
from 10% to 50% by weight of the composition of a  
5 halogen donor component;  
an effective amount of a halogen volatilisation agent;  
and  
a char-inducing component which induces formation of a  
char upon application of flame to the composition.

10

2. A capstock polymer composition according to claim 1,  
wherein the acrylic polymer component comprises from 40 to 75%  
by weight of the composition.

15 3. A capstock polymer composition according to claim 1 or  
claim 2, wherein the acrylic polymer component comprises at  
least a major amount of a high molecular weight acrylic  
polymer that has a molecular weight of from about 100,000 to  
about 500,000.

20

4. A capstock polymer composition according to claim 3,  
wherein the acrylic polymer component comprises more than 50%  
by weight, based on the weight of the acrylic component, of a  
high molecular weight acrylic polymer having a molecular  
25 weight in the range of from about 150,000 to about 350,000, on  
a number average basis, and up to 50% by weight, based on the  
weight of the acrylic polymer component, of a low molecular  
weight acrylic polymer having a molecular weight of from about  
10,000 to about 100,000, on a number average basis.

30

5. A capstock polymer composition according to any one of  
claims 1 to 4, further comprising a pigment for imparting

colour to the material.

6. A capstock polymer composition according to any one of claims 1 to 5, wherein the halogen donor component comprises a halogen-containing polymer which has a K value of from about 50 to about 65.

7. A capstock polymer composition according to any one of claims 1 to 6, wherein the halogen donor component comprises polyvinyl chloride.

10

8. A capstock polymer composition according to any one of claims 1 to 7, wherein the char-inducing component comprises a blend of vitreous materials which exhibits a broad melting range of from about 350°C up to about 800°C and which devitrifies at temperatures in the range of from about 800° to about 900°C.

9. A capstock polymer composition according to any one of claims 1 to 7, wherein the char-inducing component comprises zinc borate, zinc stannate, or a mixture thereof.

10. A capstock polymer composition according to any one of claims 1 to 9, wherein the amount of the char-inducing component ranges from about 2% by weight up to about 15% by weight of the composition.

11. A capstock polymer composition according to any one of claims 1 to 10, comprising a blend of:

from 40 to 75% by weight of the composition of a melt extrudable acrylic polymer component;

from 10% to 30% by weight of the composition of a polyvinyl chloride which has a K value of from about 50 to

about 65 as a halogen donor component;

from 3% to 8% by weight of the composition of sodium antimonate or antimony trioxide as a halogen volatilisation agent; and

from 2% to 15% by weight of the composition of at least one zinc salt selected from zinc stannate and zinc borate as a char-inducing component.

12. A polymer laminate comprising a base material and a coat of a capstock polymer composition according to any one of claims 1 to 11.

13. A polymer laminate according to claim 12, wherein the coat has a thickness of from about 40  $\mu\text{m}$  to about 500  $\mu\text{m}$ .

14. A polymer laminate according to claim 12 or claim 13, wherein the base material comprises polyvinyl chloride.





## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>6</sup> :</b> <b>C08L 33/06, C08K , B32B , C09D , C08J</b>	<b>A2</b>	<b>(11) International Publication Number:</b> <b>WO 00/37557</b> <b>(43) International Publication Date:</b> 29 June 2000 (29.06.00)
<b>(21) International Application Number:</b> PCT/GB99/04353 <b>(22) International Filing Date:</b> 22 December 1999 (22.12.99) <b>(30) Priority Data:</b> 98310616.2 22 December 1998 (22.12.98) EP <b>(71) Applicant (for all designated States except US):</b> H. B. FULLER COATINGS LTD. [GB/GB]; 95 Aston Church Road, Nechells, Birmingham B7 5RQ (GB). <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> WELTON, Nicholas, Jason [GB/GB]; 29 Cranhill Close, Solihull, West Midlands B92 8RX (GB). DODD, Keith, Herbert [GB/GB]; 5 Parson's Drive, Richmond Park, Gnosall, Staffordshire ST20 0QS (GB). <b>(74) Agent:</b> EYLES, Christopher, Thomas; W. P. Thompson & Co., Celcon House, 289-293 High Holborn, London WC1V 7HU (GB).		<b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>Without international search report and to be republished upon receipt of that report.</i>
<b>(54) Title:</b> CAPSTOCK POLYMER COMPOSITION  <b>(57) Abstract</b>  The invention relates to a capstock polymer composition comprising a blend of a melt extrudable acrylic polymer component; from 10% to 50% by weight of a halogen donor component; a halogen volatilisation component; and a char-inducing component which induces formation of a char upon application of flame to the composition.		

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## CAPSTOCK POLYMER COMPOSITION

Field of the Invention

The present invention relates to a polymer composition for use as a capstock material for making plastic laminates.

5 Background of the Invention

Low cost plastics, such as poly(vinyl chloride) (PVC), have many commercial uses but often require capping with other materials if the intended use is likely to prompt unreasonably rapid deterioration of the base plastic or if it is desired to impart colour, gloss, weatherability or some other property to the base plastic.

10 For example PVC is suitable, because of its low cost, rigidity, ease of processing (for example, by extrusion) and other mechanical properties, for use in a variety of building materials and products. Examples are window systems, including profiles for all types of windows, door sets and associated items; roofline and cladding products, such as fascia, bargeboards, soffits, shiplap and vee groove cladding; rainwater systems, such as guttering, downpipes and accessories; and piping systems, such as waste water and process piping for use above and below ground. Often such products utilise expanded PVC so as to save material and reduce the weight of such products. However, PVC is relatively unstable to heat and light and does not have good durability or resistance to weathering. This may present particular problems when the appearance of the building product, such as a window frame, is important. Deterioration of PVC involves yellowing of the material and may be unsightly. Similarly, the problem of poor weatherability may be of particular concern in, for example, guttering. Deterioration of the PVC guttering may lead to cracks in

the material which may ultimately allow egress of water from the guttering.

Other base plastics which may be used in place of, or as well as, PVC in such applications include polybutene, polypropylene, polybutylene terephthalate (PBT), polyamides, and styrene-based polymers, such as acrylonitrile-butadiene-styrene copolymers, high impact polystyrene, and styrene-acrylonitrile copolymers. Such materials may be foamed for cost or weight considerations. These materials generally have one or more of PVC's disadvantages.

One conventional solution to the problem of low durability of the base plastic is to coat the plastic with a thin layer of a capstock material, i.e. another material having better resistance to weathering and perhaps other desirable properties such as colour or gloss. Such capstock materials are known in the art.

United States Patent No. 3,458,391 discloses a laminated article which comprises in superimposed relationship a rigidity imparting base member having bonded thereto a protective film of a blend of from 90% to 40% by weight of poly (methyl methacrylate) (PMMA) or poly (ethyl methacrylate) and from 10% to 60% by weight of poly (vinylidene fluoride).

United States Patent No. 5,322,899 teaches a capstock polymer composition comprising 30 to 80% of a fluoropolymer component, 5 to 50% of poly (methyl methacrylate) and 5 to 50% of poly (ethyl methacrylate).

International Patent Application No. WO93/05959 discloses a plastic composite comprising an extrusion product having at least two layers including a layer of a first polymeric substance comprising from 1 to 70% by weight of an acrylic material, a fluoropolymer and an

inorganic pigment in contact with a second polymeric layer.

Another capstock material containing polyvinylidene fluoride is disclosed in European Patent Specification No. 0151812 A2.

5 United States Patent No. 5,132,164 also discloses a fluorine resin type film having a multi-layer structure.

Coextrusion methods for producing composite polymers are also known, as described for example in British Patent Specification No. 2071007 A.

10 Thermoforming a plastic substrate with simultaneous formation of an integral coating on a surface of the thermoplastic is taught in United States Patent No. 5,268,203.

15 A review of developments in plastic window frames, including PVC, coextruded and PMMA-coated profiles, PVC extrusion equipment, expandable PVC, and stabilisers can be found in Macplas Int., June 1991, pages 57 to 64. Blends of self-extinguishing PVC with high impact polystyrene, PMMA and their rheological properties are  
20 disclosed in a paper entitled "Fire Retardant Blends, Alloys and Thermoplastic Elastomers", Conference Proceedings Hilton Head Island, SC, 17<sup>th</sup> to 20<sup>th</sup> March 1991, pages 59 to 84.

25 Whilst fluoropolymers have been suggested widely as capstock ingredients because of their weatherability and chemical resistance, they are extremely expensive in relation to other polymers.

One particularly important property of capstock materials is fire retardancy. In United States Patent No.  
30 4,965,309 a smoke depressant additive for plastics is proposed which comprises an inorganic zinc compound, an inorganic magnesium compound other than magnesium oxide,

an inorganic molybdenum compound, and an inorganic copper compound. Incorporation of such a smoke depressant additive in PVC is proposed.

Japanese Unexamined Patent Specification No.

54/034359 discloses a composition which comprises 100 parts by weight of a polymer mixture consisting of 95-35 parts by weight polytetramethylene terephthalate and 5-65 parts by weight of a methacrylate polymer containing at least 50 weight % of a polymer of methyl methacrylate, 3-40 parts by weight of a halogen compound, such as a bisphenol-A type brominated epoxy resin, 1-30 parts by weight of an antimony compound, such as antimony trioxide, and 3-70 weight % (of total resin) of non-electrically conductive inorganic filler, such as glass fibre and talc.

I. N. Razinskaya et al., Plast Massy, No. 2 (1983), pages 49 to 50, investigated synergism with the simultaneous action of three fireproofing agents, i.e. antimony trioxide, trichloroethylphosphate and polyvinylchloride, in compositions with methyl methacrylate.

Arakawa et al., Kobunshi Ronbunshu (Japanese Polymer Science and Technology), Vol. 31, No. 10 (1974), pages 643 to 647, made a quantitative study of the synergetic effect of chlorine and phosphorus or antimony in conferring self-extinguishing properties on poly(methyl methacrylate), using a blend of poly(methyl methacrylate) and a vinyl chloride-vinylidene chloride copolymer.

While acrylic polymers, such as poly(methyl methacrylate), have many of the properties desired in a capstock material, their flammability is a drawback to their use. This drawback can be mitigated, to some extent, by formulating such acrylic polymer capstocks as coextrudates with halogen-donating materials, for example

a halogen-containing polymer such as PVC. The halogen-donating polymer provides a source of halogen atoms which can act as a chain breaker in a free radical combustion reaction, in the event of the capstock being exposed to fire. If the base plastic is PVC and the halogen-donating polymer is PVC, a strong bond is formed between identical ingredients of the base plastic and the capstock. However, in order to meet stringent fire safety regulations in many countries, it will often be necessary to include quite large quantities (typically at least about 70% by weight) of halogen-donating polymer in the capstock. Whilst the incorporation of large amounts of, for example, PVC in the composition may have an improving effect upon the fire retardance of the capstock, it may also lead to increased deterioration of the capstock on exposure to U.V. light or other adverse environmental conditions.

It is particularly important for many building materials and products to be resistant to the spread of flame. Ideally, polymer composites for use in such materials and products should meet the standards of BS476 Part 7 in the United Kingdom and should pass corresponding fire safety tests in other countries.

Accordingly, there is a need for capstock materials which exhibit fire retardance, good weatherability and durability, which are cost effective to manufacture, which have good appearance, and which may include colour.

It is therefore an object of the present invention to provide a polymer capstock which is fire retardant and which meets one or more of these other needs.

#### Summary of the Invention

According to the present invention, there is provided a capstock polymer composition comprising a blend

of:

a melt extrudable acrylic polymer component;  
from 10% to 50% by weight of the composition of a  
halogen donor component;

an effective amount of a halogen volatilisation  
agent; and

a char-inducing component which induces formation of  
a char upon application of flame to the composition.

Detailed Description of the Invention

The invention provides a capstock polymer  
composition. This comprises a melt extrudable acrylic  
polymer component that preferably consists of or comprises  
an acrylic polymer that has a molecular weight of from  
about 50,000 to about 500,000 on a number average basis.  
The capstock polymer composition further comprises from  
about 10% to about 50% by weight of the composition of a  
halogen donor polymer component, such as poly(vinyl  
chloride) (PVC), which is adapted to behave as a halogen  
donor upon application of a flame to the composition. A  
third ingredient of the composition is a halogen  
volatilisation agent, such as antimony trioxide or sodium  
antimonate, while a fourth component is a char-inducing  
component, that is to say a component that upon  
application of flame to the composition induces formation  
of a char upon the surface of the composition and thereby  
formation of a barrier to penetration of the flame to  
subjacent material. Typical char-inducing components  
include vitrifying ceramics and zinc salts, such as zinc  
stannate, zinc borate, or a mixture thereof.

In a preferred embodiment of the invention, the melt  
extrudable acrylic polymer component comprises at least a  
major amount of a high molecular weight acrylic polymer  
that has a molecular weight of from about 50,000, more



preferably about 100,000, to about 500,000, even more preferably from about 150,000 to about 350,000, on a number average basis. Such acrylic resins can be formed by polymerisation of esters or amides of acrylic acid or of an  $\alpha$ -alkylacrylic acid, for example methacrylic acid. Examples of suitable esters include acrylamide,  $\alpha$ -methylacrylamide, methyl acrylate, ethyl acrylate, methyl methacrylate, ethyl methacrylate, and the like. Besides homopolymers, random or block copolymers can also be used. Preferred high molecular weight materials include Oroglas DRT and Oroglas HFI-10 which are available from Atohaas UK Ltd., of 1 Focus Park, Ashbourne Way, Cranmore Boulevard, Shirley, Solihull B90 4QU, England and Diakon, which is available from ICI Acrylics, P.O. Box 90, Wilton Centre, Middlesbrough, Cleveland TS90 8JE, England.

The melt extrudable acrylic polymer component preferably comprises more than about 50% by weight of a high molecular weight acrylic polymer and up to about 50% by weight of a low molecular weight acrylic polymer, having, for example, a molecular weight in the range of from about 10,000 to about 100,000 on a number average basis. Typical low molecular weight acrylic polymers include Paraloid B-67 which is available from Rohm & Haas, of Lennig House, 2 Masons Avenue, Croydon CR9 3NB, England. Preferably, the total melt extrudable acrylic polymer component comprises from about 40 to about 75% by weight of the capstock polymer composition.

The capstock polymer composition of the invention incorporates from 10% to 50% by weight of the composition, preferably 10% to 30% by weight of the composition, of a halogen donor component, for example PVC, as a flame retardant. Although halogen donor polymers have good flame retarding properties, they also tend to be unstable

to U.V. light. Accordingly, prior art blends of PVC and low molecular weight acrylic polymers which include relatively high proportions typically about 70% by weight or more of PVC, tend to degrade upon prolonged exposure to UV light. In the capstock polymer composition of the invention, the proportion of halogen donor component is reduced relative to the prior art blends. The relatively low proportion of halogen donor component, in combination with the use of high molecular weight acrylic polymers, confers excellent U.V. stability on the capstock polymer composition of the invention.

The halogen-donor component is preferably PVC, but other suitable halogen donor polymers include halogen-containing polymers such as chlorinated polyethylene, fluoropolymers, e.g. polyvinylidene fluoride and polytetrafluoroethylene, polyvinylidene chloride, and brominated polyacrylates. Other halogen donors that can be mentioned are hexabromocyclododecane, octabromodiphenyl ether and Cereclor (which is available from Imperial Chemical Industries plc).

If the halogen donor component is PVC, then it preferably has a K value of from about 50 to about 65. A suitable commercial material is Evipol SH5730 available from EVC Limited of 1 King's Court, Manor Farm Road, Runcorn, Cheshire WA7 1HR, England or Benvic ER1525/1A available from Solvay Chemicals, Grovelands Business Centre, Boundary Way, Hemel Hempstead, Hertfordshire HP2 7TE, England.

The capstock polymer composition further comprises an effective amount, typically from about 1% by weight up to about 10% by weight of a halogen volatilisation agent whose function is to combine with halogen radicals released by the halogen donor upon application of a flame

to the surface of the capstock polymer composition and from a volatile halide by reaction therewith. Examples of suitable halogen volatilisation agents include antimony oxide, antimonate salts such as an alkali metal antimonate, e.g. sodium antimonate, and stannate salts such as zinc stannate, or a mixture of two or more thereof. Preferably the capstock polymer composition contains from about 3% by weight up to about 8% by weight of halogen volatilisation agent. Halogen radicals released by the halogen donor, in the presence of a flame, combine with, for example, antimony oxide to form the volatile antimony trichloride,  $\text{Sb}_2\text{Cl}_6$ , it is postulated. In the flame this dissociates to form halogen radicals again which react with hydroxy radicals which are believed to be the radicals which are principally responsible for spread of a flame. By trapping the hydroxy radicals, the free radical chain reaction is terminated, thus helping to prevent further spread of flame.

In a preferred composition a synergistic combination of PVC and antimony oxide (or sodium antimonate) is used which forms volatile radical scavengers which terminate the flame propagation step in the burning process. The ratio of PVC to antimony oxide (or sodium antimonate) must be maintained for efficient use of the halogens. The presence of PVC reduces the UV resistance of the capstock. The heat and shear sensitive nature of the polymer necessitates addition of PVC to the capstock polymer composition at a relatively low temperature (e.g. less than about  $200^\circ\text{C}$ ) so as to avoid the danger of a highly UV sensitive material being produced due to the formation of hyper-sensitive radicals caused by dehydrochlorination of the polymer backbone.

The char-inducing component is typically present in

an amount of from about 2% to about 15% by weight of the capstock polymer composition. It may be a vitrifying ceramic material, such as Ceepree Microfine, which is a product of Brunner Mond Limited available from Ceepree Products of 135a Linaker Street, Southport, Merseyside PR8 5DF, England. Other suitable char-inducing components are zinc borate, zinc phosphate, zinc stannate, and phosphonates, such as dimethyl methylphosphonate.

Other ingredients may be incorporated in the capstock polymer composition of the invention, typically in amounts not exceeding about 5% by weight of the capstock polymer composition. For example, a PVC stabiliser, such as a carboxylated organotin available from Ciba Geigy Limited of Hulley Road, Macclesfield, Cheshire SK10 2NX, England under the designation Irgastab T-9 or epoxidised soya bean oil available from Ciba-Geigy Limited under the designation Irgaplast-39 may be used.

A heat sink material is another example of a minor optional ingredient. Such heat sink materials include alumina trihydrate and magnesium hydroxide.

As an example of another minor ingredient small quantities of fluoropolymers such as polytetrafluoroethylene (PTFE) may be used to improve the processability of the material during manufacturing. One suitable commercial material is Lancowax TF1778 available from Capricorn Chemicals of Lisle Lane, Ely, Cambridgeshire CB7 4AS, England. An absorber of ultra-violet light, such as the benzotriazol Tinuvin 320 available from CIBA Geigy Limited or Lowilite 55, available from Great Lakes, P.O. Box 44, Oil Sites Road, Ellesmere Port, South Wirral, L65 4GD, England, may also be incorporated as a minor ingredient in the capstock polymer composition.

The capstock polymer composition of the invention

may further comprise a pigment or mixture of pigments for imparting colour to the composition. Typically the amount of pigment or mixture of pigments does not exceed about 10% by weight of the composition, more usually from about 0.5% to about 5% by weight of the composition. Suitable pigments include those set out in the following Table 1.

Table 1

Chemical Name	Trade Mark	Supplier Name & Address
Iron Oxide	Bayferrox 130B	Bayer plc, Strawberry Hill, Newbury, Berkshire RG14 1JA, England
Iron Oxide Black	Bayferrox 303T	Bayer plc, Strawberry Hill, Newbury, Berkshire RG14 1JA, England
Carbon Black	Black Pearls 900	Cabot Carbon Ltd., Lees Lane, Stanlow, Ellesmere Port, South Wirral L65 4HT, England
Azo Condensation Product	Chromophtal Brown 5R	Ciba Specialty Chemicals, Hulley Road, Macclesfield, Cheshire SK10 2NY, England
Iron Oxide	Colortherm 20	Bayer plc, Strawberry Hill, Newbury, Berkshire RG14 1JA, England
Phthalocyanine	Heliogen Green L8730	BASF plc, Cheadle Hulme, Cheshire SK8 6QG, England
Iron Chrome Spinel	Irgacolor Black 10399	Ciba Specialty Chemicals, Hulley Road, Macclesfield, Cheshire SK10 2NY, England
Carbon Black	Regal Black 400	Cabot Carbon Ltd., Lees Lane, Stanlow, Ellesmere Port, South Wirral L65 4HT, England
Titanium Dioxide	Tiona RCL628	Millenium Inorganic Chemicals, P.O. Box 26, Grimsby DN41 8DP, England

In selecting a pigment for incorporation into a capstock polymer composition it is preferred to select an inorganic pigment, preferably one that has a low heat gain when exposed to sunlight.

The invention also provides a polymer laminate

comprising a base material, such as PVC, and a layer of a capstock polymer composition according to the invention. Such a polymer laminate can be used to make building materials and products, such as window systems, including profiles for all types of windows, door sets and  
5 associated items; roofline and cladding products, such as fascia, bargeboards, soffits, shiplap and vee groove cladding; rainwater systems, such as guttering, downpipes and accessories; and piping systems, such as waste water and process piping for use above and below ground. In  
10 such products the base material can be in expanded form, for example expanded PVC.

Preferably, the layer has a thickness of from about 40  $\mu\text{m}$  to about 500  $\mu\text{m}$ . For use on rigid base polymers, such as PVC, thicknesses of from about 40  $\mu\text{m}$  to about 100  
15  $\mu\text{m}$  can be used. However, a thicker layer may be desirable when the base polymer is foamed, for example foamed PVC. In this case the layer is desirably at least about 150  $\mu\text{m}$  to about 200  $\mu\text{m}$  thick in order to hide any surface blemishes due to the foamed nature of the underlying base  
20 polymer.

The capstock polymer composition of the invention has a number of important advantages over capstock materials hitherto known. The capstock polymer composition of the invention does not depend for its  
25 weatherability and U.V. stability on the use of fluoropolymers, which are very expensive and also environmentally poor materials because of the difficulty of breaking down carbon-fluorine bonds in the environment.

The use in the capstock polymer composition of the  
30 invention of high molecular weight acrylic polymers confers excellent mechanical properties, of strength and rigidity, on the capstock polymer composition of the

invention and also confers good U.V. stability on the capstock polymer compositions.

The flame retardancy of the composites of the invention is maintained, despite the relatively low proportion of halogen donor polymers therein, at an excellent level through the supplemental fire retardant effect of the char-inducing material.

Prior art acrylic/PVC blends have used relatively low molecular weight acrylic polymers. Lower molecular weight acrylic polymers have poorer mechanical properties and less U.V. stability than do the high molecular weight acrylic polymers used in this invention. Hitherto, it has not been possible to use higher molecular weight acrylic polymers because of degradation of the PVC at the melt temperature of the acrylic polymer. The invention allows high molecular weight acrylic polymers to be used without degrading the PVC. Thus the invention preferably utilises a blend of a high molecular weight acrylic polymer with a low molecular weight acrylic polymer.

A number of minor ingredients are desirably incorporated in the composition of the invention.

Further discussion of the essential ingredients of the composite is included below as part of general discussion of the preferred and minor ingredients:

**Oroglas DRT/HFI-10 - High M<sub>w</sub> Polymethylmethacrylates (PMMA's)**

These high Mw PMMA's are commercially available from Atohaas UK Limited, of 1 Focus Park, Ashbourne Way, Cranmore Boulevard, Shirley, Solihull B90 4QU, England and confer good mechanical properties and excellent UV stability on the composite materials of the invention, as well as having good adhesion to the base plastic, for example PVC.

**Paraloid B-67 Low M<sub>w</sub> Poly(iso-butyl methacrylate) (IBMA)**

This low M<sub>w</sub> acrylate resin has a T<sub>g</sub> around 50°C. It may be used to improve the processibility of the Oroglass resins by reducing the T<sub>g</sub> of the overall system.

**Microfine Ceepree**

5 This intumescent ceramic powder is commercially available from Ceepree Products, of 135a Linaker Street, Southport, Merseyside PR8 5DF, England and vitrifies upon the action of intense heat to form an oxygen barrier layer on the surface of the material. This action, along with  
10 the effect of the radical scavenging polymer renders the capstock material of the invention incapable of supporting a flame.

**Pigment**

15 High durability pigments may be used to maintain good colour retention properties of the capstock under the action of UV light. To impose low heat gain properties to the material highly IR reflecting or low IR absorbing pigments are used, for example one or more of the pigments listed in Table 1 above.

**Irgastab T-9/Irgaplast 39**

20 These primary and secondary, respectively, PVC heat stabilisers are used to limit the formation of active radical centres on the PVC by limiting their propagation (T-9) and deactivating the autocatalytic HCl produced (T-  
25 39).

**Lancowax TF 778**

This material is a PTFE wax used a process aid to help the mixed melt material flow through the extrusion die in the manufacturing process.

**Tinuvin 320**

30 This material is a UV absorber which helps to protect the PVC in the capstock of the invention as well



as preventing transmission of the UV to the base plastic when coated with a capstock material according to the invention.

**Tinuvin 770**

This is a hindered amine light stabiliser (HALS).

**Uniwax 1760**

This is a polyethylene wax lubricant.

A particularly preferred capstock polymer composition according to the invention comprises a blend of:

from 40 to 75% by weight of the composition of a melt extrudable acrylic polymer component;

from 10% to 30% by weight of the composition of a polyvinyl chloride which has a K value of from about 50 to about 65 as a halogen donor component;

from 3% to 8% by weight of the composition of sodium antimonate or antimony trioxide as a halogen volatilisation agent; and

from 2% to 15% by weight of the composition of at least one zinc salt selected from zinc stannate and zinc borate as a char-inducing component.

In order that the invention may be properly understood and fully carried into effect, a number of specific examples will now be fully described.

**Example 1**

A capstock composition according to the invention was made by blending components A and B using a screw extruder with a supplementary side feeder.

Component A

	parts by weight	
Oroglas	22.65	High Mw, high impact, PMMA
Oroglas	22.65	Med/high Mw, high impact PMMA
Paraloid B-67	10.00	Low Mw IBMA
Ceepree Microfine	10.00	Vitrifying ceramic fire barrier
Pigment	1.1	Low heat gain pigment package

Component B

	parts by weight	
Evipol SH5730	25.00	Suspension PVC(K value of 57)
Irgastab T-9	2.00	Carboxylated organotin heat stabiliser
Irgaplast T-39	2.00	Epoxidised soya bean oil, HC1 sink
Lancowax TF1778	0.20	PTFE wax
Timinox RT	6.50	Low pigmentary antimony oxide ( $\text{Sb}_2\text{O}_3$ )
Tinuvin 320	0.53	UV absorber, benzotriazol type

Component A contains the main binder, some fire barrier and the pigment, and is added in the main feed of the extruder. Due to the high viscosity of this material during the melting process a lot of shear heat is generated in the extruder. The screw is set to melt the binder material quickly and compound the inorganic additives before going through a forwarding cooling stage. This brings the melt temperature down from around 230°C to around 195°C. At this point Component B, containing the heat sensitive PVC, is added through a side feeder, along with the stabiliser package, wax and the fire synergist. After a quick mix the product is degassed under vacuum. It is then discharged through a single strand die and quenched in a water bath before being pelletized and

dried.

Example 2

The following method was used to produce a capstock composite from the following components:

Component A

Trade Name	% by weight	Chemical Name
Oroglas HFI-10	26.08	Med/High M <sub>w</sub> High Impact PMMA
Oroglas DRT	26.08	High M <sub>w</sub> High Impact PMMA
Flamtard Z10	5.00	Zinc Borate
Irgaplast 39	1.04	Epoxidised Soya Bean Oil
Uniwax 1760	1.50	Ethylene Bis-Stearamide
Pigment	2.50	Low heat gain pigment package

Component B

Trade Name	% by weight	Chemical Name
Benvic ER1525/1A	29.00	PVC Compound (K value of 64)
Sandovur VSU	0.25	Oxanilide derivative
Lowilite 55	0.25	Benzotriazole UV absorber
Pyrobloc SAP-2	5.00	Sodium Antimonate
Flamtard S	3.30	Zinc Stannate

Component A contained the main binder, some fire barrier, process aids and the pigment and was added to the main feed of a twin screw extruder. The screws were set up to impart high shear to component A. Barrel temperatures were set at 215°C and a screw speed of 250 rpm was used which gave a discharge temperature of 240°C. Once compounded, component A was pelletised and dried for 3 hours at 70°C in an air circulating drier.

The compounded granules of Component A were then

added to the pre-weighed component B, which contained the remaining shear sensitive fire retardant additives and the UV absorbers, and was compounded. The barrel temperatures were set at 180°C and a screw speed of 180 rpm was used which gave a discharge temperature of 195°C. This second  
5 extrusion used a twin screw expander fitted with screws designed to impart low shear. The compound was pelletised and then dried in an air circulating drier for a minimum of three hours at 55°C.

#### Example 3

10 Using a co-extruder set-up with exactly the same temperature profile as with standard PVC, the pelletised acrylic based capstock of Example 1 was poured into the capstock hopper of a co-extruder fitted with a coextrusion die suitable for extrusion of a PVC cladding panel having  
15 a coating of a capstock. At start up PVC was used both as base polymer a material and also as capstock. The capstock materials were changed over without a break in the running of the machine. It took 10 to 20 minutes for the coloured acrylic capstock to purge through the PVC.  
20 Then the coloured contoured PVC cladding panel which had an average capstock layer thickness of about 300 µm was cut to the correct length. The cladding was then ready for installation. It was subjected to BS476: Part 7: 1987 Method for Classification of the Surface Spread of Flame  
25 of Products and achieved Class 1Y.

#### Example 4

The general method described in Example 2 was used to produce a capstock from the following components:

Component A

Trade Name	% by weight	Chemical Name
Orogas HFI-10	26.48	Med/High M <sub>w</sub> High Impact PMMA
Orogas DRT	26.48	High M <sub>w</sub> High Impact PMMA
Flamtard Z10	5.00	Zinc Borate
Irgaplast 39	1.04	Epoxidised Soya Bean Oil
Uniwax 1760	1.00	Polyethylene wax lubricant
Licowax E	0.50	Montan wax
Pigment	2.50	Low heat gain pigment package

Component B

Trade Name	% by weight	Chemical Name
Benvic ER1525/1A	29.00	PVC Compound (K value of 64)
Pyrobloc SAP-2	6.50	Sodium Antimonate
Sandovur VSU	0.50	Oxanilide derivative
Tinuvin 770	0.50	HALS
Irgaplast 39	0.50	Epoxidised soya bean oil

Example 5

0.5 m x 0.5 m PVC panels were produced by the general procedure described in Example 3 using the capstock compositions of Examples 1, 2 and 4 respectively. These panels were subjected to a fire resistance test similar to that described in BS476: Part 7: 1987 Method for Classification of the Surface Spread of Flame of Products. All three panels exhibited similarly good performances in this test.

CLAIMS:

1. A capstock polymer composition comprising a blend of:  
a melt extrudable acrylic polymer component;  
from 10% to 50% by weight of the composition of a  
5 halogen donor component;  
an effective amount of a halogen volatilisation agent;  
and  
a char-inducing component which induces formation of a  
char upon application of flame to the composition.

10

2. A capstock polymer composition according to claim 1,  
wherein the acrylic polymer component comprises from 40 to 75%  
by weight of the composition.

15 3. A capstock polymer composition according to claim 1 or  
claim 2, wherein the acrylic polymer component comprises at  
least a major amount of a high molecular weight acrylic  
polymer that has a molecular weight of from about 100,000 to  
about 500,000.

20

4. A capstock polymer composition according to claim 3,  
wherein the acrylic polymer component comprises more than 50%  
by weight, based on the weight of the acrylic component, of a  
high molecular weight acrylic polymer having a molecular  
25 weight in the range of from about 150,000 to about 350,000, on  
a number average basis, and up to 50% by weight, based on the  
weight of the acrylic polymer component, of a low molecular  
weight acrylic polymer having a molecular weight of from about  
10,000 to about 100,000, on a number average basis.

30

5. A capstock polymer composition according to any one of  
claims 1 to 4, further comprising a pigment for imparting

colour to the material.

6. A capstock polymer composition according to any one of claims 1 to 5, wherein the halogen donor component comprises a halogen-containing polymer which has a K value of from about 50 to about 65.

7. A capstock polymer composition according to any one of claims 1 to 6, wherein the halogen donor component comprises polyvinyl chloride.

10

8. A capstock polymer composition according to any one of claims 1 to 7, wherein the char-inducing component comprises a blend of vitreous materials which exhibits a broad melting range of from about 350°C up to about 800°C and which devitrifies at temperatures in the range of from about 800° to about 900°C.

9. A capstock polymer composition according to any one of claims 1 to 7, wherein the char-inducing component comprises zinc borate, zinc stannate, or a mixture thereof.

10. A capstock polymer composition according to any one of claims 1 to 9, wherein the amount of the char-inducing component ranges from about 2% by weight up to about 15% by weight of the composition.

11. A capstock polymer composition according to any one of claims 1 to 10, comprising a blend of:

from 40 to 75% by weight of the composition of a melt extrudable acrylic polymer component;

from 10% to 30% by weight of the composition of a polyvinyl chloride which has a K value of from about 50 to

about 65 as a halogen donor component;

from 3% to 8% by weight of the composition of sodium antimonate or antimony trioxide as a halogen volatilisation agent; and

from 2% to 15% by weight of the composition of at least one zinc salt selected from zinc stannate and zinc borate as a char-inducing component.

12. A polymer laminate comprising a base material and a coat of a capstock polymer composition according to any one of 10 claims 1 to 11.

13. A polymer laminate according to claim 12, wherein the coat has a thickness of from about 40  $\mu\text{m}$  to about 500  $\mu\text{m}$ .

15 14. A polymer laminate according to claim 12 or claim 13, wherein the base material comprises polyvinyl chloride.



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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/GB99/04353 <b>(22) International Filing Date:</b> 22 December 1999 (22.12.99) <b>(30) Priority Data:</b> 98310616.2 22 December 1998 (22.12.98) EP <b>(71) Applicant (for all designated States except US):</b> H. B. FULLER COATINGS LTD. [GB/GB]; 95 Aston Church Road, Nechells, Birmingham B7 5RQ (GB). <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> WELTON, Nicholas, Jason [GB/GB]; 29 Cranhill Close, Solihull, West Midlands B92 8RX (GB). DODD, Keith, Herbert [GB/GB]; 5 Parson's Drive, Richmond Park, Gnosall, Staffordshire ST20 0QS (GB). <b>(74) Agent:</b> EYLES, Christopher, Thomas; W. P. Thompson & Co., Celcon House, 289-293 High Holborn, London WC1V 7HU (GB).		<b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i> <b>(88) Date of publication of the international search report:</b> 8 September 2000 (08.09.00)
<b>(54) Title:</b> CAPSTOCK POLYMER COMPOSITION  <b>(57) Abstract</b>  The invention relates to a capstock polymer composition comprising a blend of a melt extrudable acrylic polymer component; from 10% to 50% by weight of a halogen donor component; a halogen volatilisation component; and a char-inducing component which induces formation of a char upon application of flame to the composition.		

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# INTERNATIONAL SEARCH REPORT

Int. Application No

PCT/GB 99/04353

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C08L33/06 C08K11/00 B32B27/18 //(C08L33/06,57:08)

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

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IPC 7 C08L C08K B32B C09D C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	<p>DATABASE WPI Section Ch, Week 7917 Derwent Publications Ltd., London, GB; Class A14, AN 79-32158B XP002104164 &amp; JP 54 034359 A (MITSUBISHI RAYON CO LTD) , 13 March 1979 (1979-03-13) abstract</p>	1,2
X	<p>RAZINSKAYA ET AL: "study of the synergism of fireproofing agents" PLAST MASSY, no. 2, 1983, pages 49-50, XP002104162 figures; tables</p>	1
	-/-	

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	<p>ARAKAWA, ABE, IMAI: "effect of fire-retardant elements" KOBUNSHI RONBUNSHU (JAPANESE POLYMER SCIENCE AND TECHNOLOGY)., vol. 31, no. 10, 1974, pages 643-7, XP002104163 TOKYO JP figure 3; table 3</p>	1

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Information on patent family members

Inter: ☐ National Application No

PCT/GB 99/04353

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		JP 60017308 B	02-05-1985